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LUBRICANT FOR COLD AND HOT FABRICATIONS OF METAL TUBE
[Kinzoku-kan=no Reikan oyobi Onkan Kakou-you Junkatsuzai]

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SPECIFICATION /605*

1. Title

Lubricant for cold and hot fabrications of metal tubes

2. Claims

(1) A lubricant agent for cold and hot processes of a metal tube, comprising:

hydrogen carbonate in the range from 5 to 55 wt%,

a dispersing agent in the range from 0.1 to 1.0 wt%, and

a surfactant in the range from 1 to 2 wt%.

(2) A lubricant agent for cold and hot processes of a metal tube, comprising:

hydrogen carbonate in the range from 5 to 55 wt%,

a dispersing agent in the range from 0.1 to 1.0 wt%,

a surfactant in the range from 1 to 2 wt%, and

metallic soap and/or graphite less than 12 wt%.

3. Detailed Explanation of the Invention

The present invention relates to a lubricant which is used for cold and hot processes of metal tubes, and in particular, it relates to an aqueous dispersion which contains hydrogen carbonate as the principal component, appropriate amounts of a dispersion agent and a surfactant, and additionally a solid lubricant material such as metallic soap and graphite.

Various metal tubes including a steel tube require various types of lubricants for improving the product quality and reducing tool abrasion

* Numbers in the margin indicate pagination in the foreign text.

(or tool burning to stick to the product surface) in processes including their cold and hot rolling, forging, extrusion, and extrusion for elongation. However, well-known lubricants do not satisfy all requirements for lubrication, being easy to remove after work, and low pollution of liquid waste.

For example, lubricants used for light machine work are metallic soap, animal/vegetable oils, mineral oil, oil for processing plastic (containing additives for extreme pressure). However, as the degree of processing increases, the interfacing area between a metal tube and a tool also increases, causing more damage to both the tool and the tube. In order to reduce the damage, a conventional method for improving the lubrication is a chemical coating film formed on the surface of the metal tube and the simultaneous use of a chemical metallic soap. The chemical coating film includes a phosphate film (for plain carbon steel and low alloy steel), an aluminum fluoride film (for aluminum), and a phosphate film (for stainless steel). In this method, there is a chemical coating film between a tube to be processed and the metallic soap film, and the lubricant film shows extremely strong adhesion with the chemical coating film, providing adequate lubrication even if the process job /606
increases. However, the effect of lubrication is degraded as the process involves more jobs. For example, the combination of the phosphate film and the chemical metallic soap loses the lubrication effect at a temperature near 350 °C. Furthermore, because the lubrication is a chemical reaction type, the management of post-process liquid waste requires special care, and the lubrication liquid needs to be replaced frequently

due to its short life time. Therefore, it brings up the issue of cost effectiveness and pollution. There is another issue of the removal of the lubrication film coated on a metal tube after the completion of a process, and it is difficult because the lubricant film often adheres to the tube too well.

Therefore, it is desirable to develop a lubricant which has a superior lubrication effect for hot and cold processes, may be handled and removed easily, and causes no waste liquid pollution.

Considering the above technological issues, the inventors of the present invention have diligently conducted research and development on lubricants that fulfill the above requirements. As a result, the inventors have discovered that an aqueous dispersive lubricant whose major component is hydrogen carbonate added small amounts of a dispersing agent and a surfactant and an optional solid lubricant is an excellent lubricant that satisfies the aforementioned requirements, and therefore, realizes the present invention. Namely, a lubricant of the present invention applied for cold and hot processes of a metal tube is an aqueous dispersing liquid, containing hydrogen carbonate in the range from 5 to 55 wt%, a dispersing agent in the range from 0.1 to 1.0 wt%, a surfactant in the range from 1 to 2 wt%, and optionally metallic soap or graphite less than 12 wt%.

As described above, a lubricant for processing a metal tube of the prior art is acceptable when the process steps are a few. However, it does not show adequate lubrication when the process involves more job steps, and loses lubrication significantly when the process temperature is high. The inventors of the present invention have investigated

lubrication of many substances at high pressure, and discovered that hydrogen carbonate such as sodium hydrogen carbonate and potassium hydrogen carbonate may form a very good lubrication film even at high pressure. Furthermore, the lubricant film may maintain high lubrication under various process conditions, i.e., it is not only stable in a cold process but also stable in a hot process at a temperature of approximately 400 °C. The stability at high temperatures may prevent a tool used in the hot process from being burnt to adhere to the tube product, and at the same time, may improve the product surface condition and prolong the life time of the machining tool. The lubricant film may be removed easily from the product surface, and its waste liquid causes little pollution. Nevertheless, hydrogen carbonate is solid, which is extremely difficult to use as a lubricant, and thus, requires an appropriate material condition. Small amounts of a dispersing agent and a surfactant added to hydrogen carbonate and dispersed in water fulfills the requirement. As the preferable amount of hydrogen carbonate is shown below, in many cases, its mixing ratio to the total amount of the lubricant agent is substantially high, exceeding the solubility for water. In other words, the aqueous hydrogen carbonate alone is an emulsion, and its stability is poor and not practically usable. However, if the aqueous suspension coexists with small amounts of a dispersing agent and a surfactant, the mixing and dispersion properties are improved and the stability of the dispersing liquid is also improved. Furthermore, the lubricant coating film may be formed more easily and the lubrication of the coated film is also promoted. The surfactant reduces the interfacial tension between

water and the solid component (i.e., hydrogen carbonate), and disperses the solid component uniformly and quickly. On the other hand, because the surfactant promotes foaming the dispersing liquid, it causes various problems (such as a lack of adhesion and non-uniform adhesion of the lubricant agent) in a process, the amount of the surfactant should be minimized. The usable surfactant may be selected from various types, and the most preferable surfactant is a fatty acids amine soap such as fatty acid mono-ethanol amide and fatty acid di-ethanol amide.

The dispersing agent improves the stability of the dispersing liquid as the sedimentation inhibitor of hydrogen carbonate. On the other hand, if the dispersing agent is too much, the dispersing liquid loses fluidity, and may not adhere to the lubrication surface uniformly. Therefore, the dispersing agent should be also minimized. There are various /607
dispersing agents that have this effect, and the most preferable dispersion agents are kneaded polysaccharides, silicates, and fatty acid metal salts.

The lubricant of the present invention may fulfill the aforementioned objectives by containing the above essential 3 components with appropriate mixing ratios, and optimal small amounts of metallic soap and/or graphite enhance the protective film effect on the tool surface and prolong its life time.

Below is a description of the experimental procedure of determining the mixing ratio.

Aqueous dispersive liquids of several different mixing ratios of hydrogen carbonate ($NaHCO_3$ and $KHCO_3$), 0.2% of linear polysaccharide as the dispersing agent, and 1.0% of fatty acid mono ethanol are prepared

and their lubrication for rolling a tube was investigated. The process condition is listed in Table 1.

Table 1

Steel type Original tube size Rolling size Cross section processing rate		SUS304 60 mm^{Φ} X 7 mm^{t} 30 mm^{Φ} X 2.9 mm^{t} 79%
Processing rate	Stroke	9.5 stokes/min
	Feeding	17 mm/stroke
	Conveying	1.615 mm/min

Table 2 shows the result of the tests.

Table 2

$NaHCO_3$ (or $KHCO_3$) containing ratio (wt%)	0	5	10	20	30	40	50	55	60
Process result	X	○	○	◎	◎	◎	◎	◎	◎

X: A tool is burnt to adhere to the tube

○: Good

◎ : Very good

From the above result, it is clearly observed that the prepared aqueous dispersive liquids of hydrogen carbonate, containing small amounts of a dispersing agent and a surfactant, show very good lubrication. In order to obtain effective lubrication of hydrogen carbonate, it is necessary to mix hydrogen carbonate at least 5%, and preferably 20% or more. The experimental result shows that there is no upper limit for lubrication. On the other hand, when the hydrogen carbonate exceeds 60%, the water dispersing liquid tends to separate itself. Another experiment

is conducted to investigate the stability of the water dispersing liquid and the amount of hydrogen carbonate in it.

A dispersing agent and a surfactant similar to the above experiment are mixed with hydrogen carbonate ($NaHCO_3$) in the range from 5 to 70 % to prepare aqueous lubricant liquids (lubrication agents), which are investigated for their stability after sitting for 200 hours.

Table 3 shows the result.

$NaHCO_3$ containing ratio (wt%)	5	10	20	30	40	45	50	55	60	65	70
Separation stability lubricant agent	◎	◎	◎	◎	◎	◎	○	○	△	X	X

◎ : Excellent--- Uniformly dispersed

○: Good --- some separation at the bottom but practically no problem

[Figure 1(B)]

△ : Problematic --- Solid component is separated vertically and solidification at the lower layer [Figure 1(C)]

X: Solid component is precipitated at the bottom [Figure 1(D)]

From the above result, it is clearly seen that the lower concentrations of hydrogen carbonate show the separation stability up to approximately 45% but once the concentration exceeds 50%, the stability is degraded, and if it exceeds 55%, the separation is substantial, with a precipitated solid component appearing, which is unusable. If the concentration is 55% or less, the lubricant may be practically usable by stirring to disperse the solid component, which is slightly separated

with the concentration, immediately before use.

From the above experiments, for satisfying lubrication and the separation stability, the concentration of hydrogen carbonate is in the range from 5 to 55 %, and more preferably it is in the range from 20 to 45 %.

Based on these experimental results, the concentrations of /608 hydrogen carbonate and the dispersing agent are fixed to 30% and 0.5%, respectively to prepare several samples while varying the concentration of surfactant, which are used for investigating the correlation of the amount of surfactant with dispersion of the solid component. In this experiment, sodium hydrogen carbonate as the hydrogen carbonate, linear polysaccharide as the dispersing agent, and the fatty acid mono-ethanol amid as the surfactant are used, respectively.

Although the solid component may not be dispersed uniformly without adding the surfactant, if 1 % or more of the surfactant is added, the solid component may be separated easily (without congelation) by simply stirring it for a short time to obtains a uniform lubricant agent. However, if the concentration of the surfactant is 2 % or more, the lubricant agent foams too much to obtain uniform adhesion of the lubricant to the tube, causing sporadic adhesion. Therefore, the optimal concentration of the surfactant is in the range from 1 to 2 % of the total amount of the lubricant agent.

The next investigation is the correlation of the amount of dispersing agent (the linear polysaccharide) with the stability of the lubricant where the concentrations of hydrogen carbonate ($NaHCO_3$) and the surfactant

(fatty acid mono-ethanol amid) are fixed to 30% and 1.0 %, respectively. The hydrogen carbonate may not be dispersed uniformly without the dispersing agent, and hence no stable lubricant may be obtained in this case. On the other hand, with 0.1 % or more of the dispersing agent, the hydrogen carbonate may be dispersed very uniformly. However, as the amount of the dispersing agent increases, the fluidity of the dispersed liquid becomes low, and when the concentration of the dispersing agent exceeds 1.0%, the dispersed liquid becomes a gel, and it is no longer usable as a lubricant. Based upon the results, the concentration of the dispersing agent is preferably in the range from 0.1 to 1.0 %.

By conducting the series of experiments described above, the preferable concentrations of the essential components, hydrogen carbonate, the dispersing agent, and the surfactant, are determined. Furthermore, another experiment is conducted for determining a more preferable concentration of hydrogen carbonate.

As shown in Table 4, various lubricants of different concentrations of hydrogen carbonate are prepared for testing their lubrication by conducting a test disclosed by the inventors of the present invention previously (Japanese Patent Application Kokai Publication No. 1977-68493). In this test method, a rum 4 presses a steel ball 3 which is placed on a short steel tube 1 which is coated with a lubricant and placed in dies 2 to roll the short tube 1. The performance of each lubricant is determined by observing the surface conditions of the inner wall of the short tube 1 and the surface of the ball 3, and because the test rolling is harsher than an actual rolling, it is possible to determine the

performance of the lubricant more strictly.

Table 4 lists the performance of each lubricant (i.e., scratches on the inner wall of the short tube 1 and on the surface of the ball 3), its adhered amount, stability, and actual rolling test (with the conditions listed in Table 1).

In this table, the test results are indicated by the following symbols:

◎ : Excellent O: Good

△ : Problematic (many surface scratches)

X: Poor (Too many surface scratches to use the lubricant)

Table 4

潤滑剤№		I a	I b	I c	I d	I e	I f	I g	I h	I i	I j	I k	I l
組成（重量%）	$NaHCO_3$	5	10	15	20	25	30	35	40	45	50	55	60
	$KHCO_3$	—	—	—	—	—	—	—	—	—	—	—	—
	Na_2CO_3	—	—	—	—	—	—	—	—	—	—	—	—
	分散剤	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
	界面活性剤	1	1	1	1	1	1	1	1	1	1	1	1
	水	93.8	88.8	83.8	78.8	73.8	68.8	63.8	58.8	53.8	48.8	43.8	38.8
性能評価	付着量(g/m^2)	3.0	7.0	14.0	22.9	30.0	36.8	61	109	195	348	—	—
	液の安定性	◎	◎	◎	◎	◎	◎	○	○	○	○	○	△
	鋼球表面傷	○	◎	◎	◎	◎	◎	◎	○	△	△	×	×
	短管内面傷	○	○	○	◎	◎	◎	◎	◎	◎	◎	◎	◎
	実際の圧延試験	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎

潤滑剤№		II a	II b	II c	II d	II e	II f	II g	II h	II i	II j	II k	II l
組成（重量%）	$NaHCO_3$	—	—	—	—	—	—	—	—	—	—	—	—
	$KHCO_3$	5	10	15	20	25	30	35	40	45	50	55	60
	Na_2CO_3	—	—	—	—	—	—	—	—	—	—	—	—
	分散剤	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
	界面活性剤	1	1	1	1	1	1	1	1	1	1	1	1
	水	93.8	88.8	83.8	78.8	73.8	68.8	63.8	58.8	53.8	48.8	43.8	38.8
性能評価	付着量(g/m^2)	2.8	60	12.5	17.9	33.8	45.8	66	120	200	355	—	—
	液の安定性	◎	◎	◎	◎	◎	◎	◎	◎	○	○	○	△
	鋼球表面傷	○	◎	◎	◎	◎	◎	◎	○	△	△	×	×
	短管内面傷	○	○	○	◎	◎	◎	◎	◎	◎	◎	◎	◎
	実際の圧延試験	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎

潤滑剤№		III a	III b	III c	III d	III e
組成（重量%）	$NaHCO_3$	5	10	15	20	25
	$KHCO_3$	—	—	—	—	—
	Na_2CO_3	25	20	15	10	5
	分散剤	0.2	0.2	0.2	0.2	0.2
	界面活性剤	1	1	1	1	1
	水	68.8	68.8	68.8	68.8	68.8
性能評価	付着量(g/m^2)	72.3	58.4	55.5	48.9	41.5
	液の安定性	◎	◎	◎	◎	◎
	鋼球表面傷	◎	◎	◎	◎	◎
	短管内面傷	○	○	◎	◎	◎
	実際の圧延試験	◎	◎	◎	◎	◎

Key for first 2 columns:

Lubricant No.	
Composition (wt%)	$NaHCO_3$
	$KHCO_3$
	Na_3CO_3
	Dispersing agent
	Surfactant
	Water
Performance test	Adhesion rate (g/m^2)
	Liquid stability
	Scratches on steel ball surface
	Scratches on inner wall of tube
	Actual rolling test

Dispersing agent: linear polysaccharide

Surfactant: Fatty acid mono-ethanol amid

The results of Table 4 suggests the followings:

(1) The rolling tests with the severe condition described above do not record few scratches on the inner wall of short tubes when the concentration of hydrogen carbonate in a lubricant is more than a pre-determined amount, which is the same as the actual rolling test. However, scratches on the surface of the ball appear more when hydrogen carbonate exceeds 40%. Scratches on the surface of the ball correlate with the weariness of the dies, and this test implies that the preferable upper limit of hydrogen carbonate is approximately 40%. Observing scratches on the inner wall of the tube shows that when the concentration of hydrogen carbonate increases from 15 % to 20%, the lubricant performance changes from "Good" to "Excellent," suggesting that the preferable lower limit is about 20%.

(2) The above tendency may be inferred from the amount of the lubricant agent adhered to the tube. A preliminary experiment has confirmed that the more the lubricant adhered to a tube, the more dies are burnt to adhere to the tube. When hydrogen carbonate exceeds 40%, the adhered amount of the lubricant rapidly increases, and hence the concentration of hydrogen carbonate is preferably less than 40%.

(3) The lubricant group III contains carbonate in addition to hydrogen carbonate. This case also may obtain good lubricants. However, if all hydrogen carbonate is replaced with carbonate, the separation stability and the lubrication are much degraded. Another preliminary experiment has confirmed that adding small amounts of a solid lubricant composed of metallic soap and/or graphite to the lubricant enhances, and reduces

the adherence of dies, in particular. The optimal amount of the solid lubricant is experimentally determined.

Another preliminary experiment indicates that adding solid lubricant composed of metallic soap and/or graphite to the aforementioned liquid lubricant further improves the performance of the liquid lubricant, and in particular the dies are less burnt to adhere to the product surface. Therefore, the next test is conducted to determine the preferable concentration of the solid lubricant.

The lubricants listed in Table 5 containing the same amounts of $NaHCO_3$, the dispersing agent, and the surfactant but not the solid lubricant (calcium stearate or graphite) are prepared to investigate the separation stability as the performance of lubricants following the same test method as Table 4. Table 5 summarizes the results.

Table 5

	潤滑剤№	IV								V							
		a	b	c	d	e	f	g	h	a	b	c	d	e	f	g	h
組成(重量%)	$NaHCO_3$	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30
	分散剤	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
	界面活性剤	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
	黒鉛	0	2	4	6	8	10	12	14	–	–	–	–	–	–	–	–
	ステアリン酸Ca	–	–	–	–	–	–	–	–	0	2	4	6	8	10	12	14
	水	68.8	66.8	64.8	62.8	60.8	58.8	56.8	54.8	68.8	66.8	64.8	62.8	60.8	58.8	56.8	54.8
性能評価	付着量(g/㎡)	36.8	45	53.7	63.7	77	105	150	290	36.8	58.2	89.3	105	120	180	240	350
	液の安定性	◎	○	◎	◎	○	○	○	△	◎	○	◎	◎	◎	○	○	△
	鋼球の表面傷	◎	◎上	◎上	◎上	◎上	△	×	×	◎	◎上	◎上	◎上	◎上	△	×	×
	鋼管の表面傷	◎	◎上	◎上	◎上	◎上	◎	◎	◎	◎	◎上	◎上	◎上	◎上	◎	◎	◎
	実際の圧延試験	◎	◎上	◎上	◎上	◎上	◎	◎	◎	◎	◎上	◎上	◎上	◎上	◎	◎	◎

Key for first 2 columns:

Lubricant No.	
Composition (wt%)	$NaHCO_3$ Dispersing agent Surfactant Graphite Calcium stearate Water
Performance test	Adhesion rate (g/m^2) Liquid stability Scratches on steel ball surface Scratches on inner wall of tube Actual rolling test

The dispersing agent and the surfactant: the same as before.
◎上: better than ◎ (burnt to adhere to the product, in particular)

As seen from Table 5, if optimal amounts of metallic soap and/or graphite are added, it enhances lubrication. The allowable amounts of

these solid lubricants are less than 12%, and for effectively improving lubrication, it is preferably in the range 2 to 8 %.

In addition to the separation stability and lubrication, a lubricant of the present invention should not attack a metal tube on which the lubricant agent is coated. Another experiment is conducted to investigate the weariness of the coated tube.

[Metal Attack]

Iron, copper, and aluminum plates (of length 60 mm and width 80 mm) which are polished with #240 sand paper are dipped in three different types of lubricants prepared for this test and these are left at a temperature of 110 °C for 2 hours. Table 6 shows the results.

Lubricant A: $NaHCO_3$ 30%, dispersing agent 0.2%, and surfactant 1%.

Lubricant B: $NaHCO_3$ 30%, dispersing agent 0.2%, surfactant 1%, and calcium stearate 4%.

Lubricant C: $NaHCO_3$ 30%, dispersing agent 0.2%, surfactant 1%, and graphite 4%.

Table 6

Sample plate	Iron	Copper	Aluminum
Lubricant A	◎	◎	◎
B	◎	○	◎
C	◎	○	◎

◎ : No attack at all.

○: Not much attack.

As seen from the above results, lubricants of the present invention do not attack the tube material.

The present invention is as described above, and the essential component is hydrogen carbonate dispersed in water uniformly, appropriate amounts of a dispersing agent and a surfactant, and an appropriate amount of solid lubricant as required, realizing a lubricant agent which has the following superior characteristics.

(1) The lubricant may form an excellent lubricant film under /611 harsh processing conditions, and hence it is possible to apply the lubricant to a wide variety of processes of different jobs. Adhesion of the lubricant to the product surface under processing is dramatically reduced and the product quality is improved. The life time of a tool used for the process is also prolonged.

(2) The lubricant is stable at high temperatures. It has lubrication under a process at a temperature around 400 °C, and hence it may be applied to both hot and a cold processes.

(3) Because no adhesives are used at all, and the principal component, hydrogen carbonate, is water soluble, it is easy to remove the lubricant after completing a process.

(4) Hydrogen carbonate is inexpensive and hence it is economical. It is also non-toxic, and causes no pollution from liquid waste.

(5) Because the lubricant is stable: it does not change, and has a long life time, it may be applicable to a continuous process.

(6) Although the lubricant is particularly useful to a process for a metal tube (rolling, in particular), it may also be applicable to roll or lengthen a metal sheet, a rod, and a wire as a lubricant.

4. Brief Explanation of the Figures

Figure 1 illustrates the dispersion observed in the separation stability test on the lubricant of the present invention, and Figure 2 depicts a simplified cross section of the performance test on the lubrication of the present invention.

1 … metal short tube, 2 … dies, 3 … steel ball, 4 … rum

Figure 1

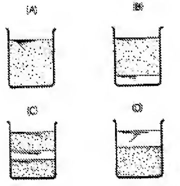

Figure 2